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(54) Title: PROCESS FOR IMPROVED HANDLING OF BITUMEN

(57) Abstract

The viscosity of bitumen, bitumen-like mixes based on polymer and bitumen and mixes of bitumen and additives is reduced by mixing said bitumen or mixes with at least one gas in a dynamic high shear mixer or in a static mixer.

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PROCESS FOR IMPROVED HANDLING OF BITUMEN

The present invention relates to a process for improved handling of bitumen. More particularly the present invention relates to a process for reducing the viscosity of bitumen, bitumen-like mixes based on polymer and bitumen and mixes of bitumen and additives.

Bitumen is used to make asphalt mixtures, bituminous membranes for roofings, emulsions for use in road making, anti-corrosion compositions, polymer-bitumen mixes, etc. In order to be handled in the mentioned applications, the bitumen is generally subjected to one of the following manipulations:

A) heating of the bitumen to such an extent that it becomes more or less liquid,
B) mixing the bitumen with solvents, or
C) emulsifying the bitumen with water.

All these three manipulations, i.e. heating, dissolving and emulsifying, have as a goal to reduce the viscosity of the initial properties after cooling, evaporation of the solvent and breaking of the emulsion and separation of the water, respectively.

The viscosity of the bitumen is expressed in Pa·s when using a dynamic viscosimeter, in mm^2/s when using capillary viscosimeters or in other units depending upon the method used in the determination of the viscosity.

Mostly, the rheology of the bitumen at about ambient temperature is described by means of the penetration test (ASTM D5) wherein at $25^{\circ}C$ a standard needle penetrates under a load of 100 g in 5 seconds into the bitumen. The penetration is expressed in tenths of a millimetre.

Another test comprises the measuring of the softening point. In this test (ASTM D36) a ring is filled with the bitumen to be tested and is heated under standard conditions at a constant rate of 5°C/min. A ball on top of the bitumen presses on the layer. When the temperature reaches a certain level, the bitumen has softened to such an extent that its deformation range has been reached. The corresponding temperature is called the ring and ball temperature (RB°) and is expressed in degrees centigrade (C°).

Each bitumen grade needs a well defined temperature or a well defined amount of solvent to reach a certain low viscosity at which it is easily handled.

The big problem, however, is that solvents cannot be used without limitations because of health and safety reasons.

Heating cannot be done in all cases without limitations either.

Thus, for instance, in road-making generation of fume limits the use of high temperatures. Also the fact that aging and hardening of the bitumen occurs in a much more dramatic way at substantially increased temperatures limits the use of heating.

Heating of a polymer-bitumen mixture to a high temperature brings the polymer in danger (depolymerisation) or destroys the homogeneity of the polymer/bitumen blend.

Heating of a hard bitumen to too high temperatures when bringing it into the form of an emulsion causes boiling of the water in the ready made emulsion.

Hence it is an object of the present invention to develop a process for reducing the viscosity of bitumen, bitumen-like mixes based on polymer and bitumen and mixes of bitumen and additives which process is not encumbered with the above mentioned drawbacks of the prior art methods based on heating, dissolving or emulsifying.

The present invention is based on the finding that when liquefied bitumen is milled through a mill together with a gas, the gas becomes finely distributed into the bitumen and as a result thereof causes a dramatic reduction of the viscosity. At the same time, the asphaltene clusters in the bitumen are more or less dispersed. Also this dispersion of asphaltene clusters causes a reduction of the viscosity.

In accordance with the above the present invention provides a process for reducing the viscosity of bitumen, bitumen-like mixes based on polymer and bitumen and mixes of bitumen and additives, which process is characterized in that said bitumen or mixes is/are mixed with at least one gas in a dynamic high shear mixer or in a static mixer.

The more intense the mixing, like high shear, and the thinner the layer between stator and rator in a dynamic mixer, the bigger is the viscosity drop and the longer will this low viscosity remain, even after hot storage of the bitumen or bitumen mix for 2-4 hours. However, as indicated above, it is also possible to use static mixers in the process of the present invention but the effects on the viscosity when using static mixers are considerably lower than those obtained by means of high shear mixers.

The gas is added to the bitumen in a continuous way at the entrance of the mill and is milled together with the bitumen.

Depending on the final use of the bitumen or mixes it is possible to select the gas in such a way that in addition to the reduction of the viscosity also other effects are achieved.

According to one embodiment of the process according to the present invention a gas is used which is chemically inert to said bitumen or mixes. The preferred gas in this connection is nitrogen.

According to another embodiment of the process according to the present invention a gas is used which causes a change in the chemical properties of said bitumen or mixes.

For instance, air can be used as the gas which leads to semiblown bitumens. Further examples of gases which causes a chemical transformations of the bitumen or mixes, are ammonia and carbon dioxide.

Using a catalyst in combination with a gas which causes a change in the chemical properties of the bitumen or mixes will have an even more intense effect on the properties of the bitumen or mixes.

The process of the present invention can be applied to all types of bitumen such as straight run bitumens, semiblown bitumens and blown bitumens of different penetration, like 5 penetration up to several hundreds - . B 1500 as an example -, bitumen-like mixes based on polymer and bitumen such as mixes with styrene-butadiene-styrene in amounts of 1 to 15 % by weight calculated on the weight of the mixture or ethylene-vinyl acetate 1-20 or others and mixes of bitumen and additives, for instance mixes of bitumen with penetrations between 5 and several hundreds and carbon black, adhesivity improvers, ageing retarders and others.

The uses of the bitumen and bitumen mixes having gas-reduced viscosity (in the following denoted "GRV-bitumen") prepared according to the invention are within the fields of making asphalt mixes, making bitumen emulsions, handling of polymer/bitumen mixes, production of roofing, anti-corrosion coating and membrane application.

The invention will now be further illustrated by means of a number of non-limitative working examples.

EXAMPLE 1

A 65 penetration bitumen, straight-run, was treated in a high shear mill from Probst und Class, Rastatt, Germany, type PUC-NA 60-RD, with nitrogen.

Temperature	:	150°C
Gap width	:	0.25 mm
Rotor speed	:	3000 rev/min
Bitumen quantity	:	0.85 l/min
Nitrogen quantity	:	not measured (free suction to open air)

The viscosity was measured at shear rate 3 sec^{-1} in a layer thickness of 1 mm - plate/plate configuration. The following results were obtained.

Temperature (C°)	Viscosity (Pa·s)	
	original	after 1 pass through mill
60	950	131
70	308	60.1
80	95.2	24.1
90	37	10.1
100	15.3	5.02
110	7.31	2.68
120	4.17	1.57
130	2.46	0.96
140	1.54	0.68

EXAMPLE 2

A 5 penetration bitumen, straight-run, was treated under the same conditions as in example 1 but at 180°C.

The viscosity was measured with CARRI-MED at shear rate 3 sec^{-1} in a layer thickness of 1 mm - plate/plate configuration. The following results were obtained.

Temperature (C°)	Viscosity (Pa·s)	
	original	after 1 pass through mill
60	1550	595
70	388	176
80	136	84
90	44.8	31.2
100	16.9	12.3
110	6.92	5.52
120	3.31	2.79
130	1.72	1.46

EXAMPLE 3

A mixture of 94 % by weight of 180/200 penetration straight-run and 6 % by weight of styrene-butadiene-styrene (Finaprene^{R} F 411 from Fina - Belgium), was treated in a high shear mill (from Probst und Class, Rastatt, Germany, type PUC-NA 60-RD) with nitrogen.

Temperature of bitumen mix	:	190°C
Bitumen quantity	:	0.380 l/min
Gap width	:	0.05 mm
Rotor speed	:	4000 rev/min
Nitrogen temperature	:	ambient
Nitrogen quantity	:	18 l/min

(lowest measurable quantity with present instruments).

The viscosity was measured with CARRI-MED at shear rate

3 sec^{-1} in a layer thickness of 1 mm - plate/plate configuration. The following results were obtained.

Temperature (C^{o})	Viscosity (Pa·s)	
	original	after 1 pass through mill
70	672	396
80	258	152
90	10.3	61.4
100	49.5	28.0
110	14.21	9.42
120	7.69	5.32
130	2.12	2.00

EXAMPLE 4

A 180/200 bitumen was treated with ammonia gas in a high shear mill (from Probst und Class, Rastatt, Germany, type PUC-NA 60-RD).

Temperature of bitumen mix	:	250°C
Quantity through mill	:	0.6 l/min
Ammonia temperature	:	ambient
Ammonia quantity	:	18 l/min
(lowest measurable quantity with present instruments)		
Gap width	:	0.05 mm
Rotor speed	:	4000 rev/min

The influence of the ammonia upon the bitumen was checked by measuring the acidity of the bitumen:

Original 180/200 bitumen : 3.69 mg KOH/g

1 x over mill : 3.51 mg KOH/g
2 x over mill : 2.68 mg KOH/g
3 x over mill : 2.54 mg KOH/g

EXAMPLE 5

A 180/200 penetration bitumen, straight-run, was treated in a static mixer (Kemics-Tubing 37-08-136, length 416 mm-21 elements from Chemineer, Derby, U.K. with air.

Penetration : original bitumen : 184
after treatment : 265

Softening point : original bitumen : 38.2°C
after treatment : 32.8°C

The viscosity was measured with CARRI-MED at shear rate 3 sec^{-1} in a layer thickness of 1 mm - plate/plate configuration. The following results were obtained.

Temperature (C°)	Viscosity (Pa·s)	
	original	after 1 pass through mill
60	88	24.2
70	24.4	8.48
80	8.3	3.35
90	3.9	1.57
100	1.6	0.83
110	0.77	0.52
120	0.42	0.32

C L A I M S

1. Process for reducing the viscosity of bitumen, bitumen-like mixes based on polymer and bitumen and mixes of bitumen and additives, c h a r a c t e r i z e d in that said bitumen or mixes is/are mixed with at least one gas in a dynamic high shear mixer or in a static mixer.

2. Process according to claim 1, c h a r a c t e r i z e d in that a gas is used which is chemically inert to said bitumen or mixes.

3. Process according to claim 1, c h a r a c t e r i z e d in that the gas is nitrogen.

4. Process according to claim 1, c h a r a c t e r i z e d in that a gas is used which causes a change in the chemical properties of said bitumen or mixes.

5. Process according to claim 4, c h a r a c t e r i z e d in that the gas is air, ammonia or carbon dioxide.

6. Process according to any of claims 4 and 5, c h a r a c t e r i z e d in that the bitumen or mix is mixed with the gas in the presence of a catalyst.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 93/00965

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: C08L 95/00, C10C 3/00, C08J 3/00
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: C08L, C10C, C08J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, CA

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 5096566 (DAWSON ET AL), 17 March 1992 (17.03.92) --	1-6
A	GB, A, 2255291 (EXXON RESEARCH AND ENGINEERING COMPANY), 4 November 1992 (04.11.92) --	1-6
A	EP, A2, 0283246 (EXXON CHEMICAL PATENTS INC.), 21 Sept 1988 (21.09.88) --	1-6

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
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INTERNATIONAL SEARCH REPORT

Information on patent family members

28/01/94

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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